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ORIGINAL ARTICLES

Sealing and Dentin Bond Strengths of Adhesive Systems

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Clinical Relevance
A perfect seal to water seems impossible with current adhesive materials.

SUMMARY

The objectives of this research were (1) to analyze the variations of the permeability of dentin after restoration with two polyacid-modified resin composites (Compoglass, Dyract) and four single-bottle adhesives (Prime & Bond 2.0, Syntac Single Component, OptiBond Solo, and Single Bond—Scotch Bond 1 in Europe—immediately (approximately 1 hour) after insertion. A perfusion system with distilled water was used at a pressure of 32.5 cm of water; (2) to study the bond strength of their interfaces; and (3) to find the correlation, if any, between both parameters. None of the materials used produced a complete cessation in fluid filtration. Tensile bond strengths were very low (maximum: P&B = 3.96 MPa) probably because of the very large bonding surfaces used (mean bonded surface area = 88.8mm²). No significant correlation was found between tensile bond strength and the sealing ability for any material.

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INTRODUCTION

The production of a perfect seal on the material-tooth interface is one of the goals of restorative dentistry in order to prevent the entrance of microorganisms and other contaminants into the environment as well as to reproduce the lost peripheral seal of dentin.

Dentin bonding agents (DBAs) are designed to produce a hermetic seal by their intimate relationship with the cut dental tissues, properly prepared, forming what is called the hybrid layer. A proper seal is required to provide clinically acceptable hydrodynamic behavior of dentinal fluid (Pashley, 1994).

Loss of dental tissues by caries or their elimination by cavity preparation tends to increase the permeability of the remaining dentin (Fogel, Marshall & Pashley, 1988; Linden, Kallsog & Wolgast, 1995).

This increase in permeability is less noticeable in coronal dentin if the smear layer is present, but becomes more noticeable in the vicinity of the pulp chamber (Tagami, Tao & Pashley, 1990). From this point of view, an ideal material would be one that lowers dentin permeability to previous levels of intact tooth (Pashley & others, 1988), regardless of the amount of remaining dentin.

Usually, the methodology employed to study the peripheral seal of the dentin-pulp complex utilizes morphological or microleakage studies in vitro or clinical studies that measure dentin sensitivity to thermal or osmotic stimuli in vivo. None of these studies takes into account the previous permeability conditions of dentin. All use subjective measuring systems such as the analogue scales for pain estimation, allow the study of only small areas of the restored interface (morphological studies), or examine microleakage using dyes. However, dyes are not normally in contact with dentin, and have different chemical and physical characteristics from the substances that would normally pass through such interfaces.

Derkson, Pashley, and Derkson (1986) described an in vitro system to measure the efficacy of sealing the dentin-pulp complex by quantification of dentinal permeability before and after obturation with different materials. This permeability is expressed by measuring the amount of fluid that comes through the area studied per unit time. This method has been used in numerous studies to determine the sealing efficacy of many materials (Pashley & others, 1985; Pashley & Depew, 1986; Del Nero, Conejo & de la Macorra, 1994, 1997; Prati & others, 1992, 1994a; Hansen, Swift & Krell, 1993; Pagliarini & others, 1996; Déjou, Sindres & Camps, 1996). A common observation in such studies was that the filtration through dentin slowed but did not stop with any of the materials studied. Similar results were found with other measuring systems based on the same idea (Terkla & others, 1987). That is, most materials do not perfectly seal immediately, although the seal improves with time in some cases.

On the other hand, there have been attempts to correlate the sealing ability of materials with the mechanical resistance of the interface they produce. There are reports about the relationship between various DBAs on different kinds of dentin (superficial, intermediate, and deep) in vivo (Pashley & others, 1993) and in vitro (McCabe & Rusby, 1992). The deepest dentin was associated with higher permeability, although the changes were not controlled. Prati and others (1994b) measured the changes in hydraulic conductivity of dentin when different DBAs were applied, but the changes in the permeability after obturation with the corresponding composite resin were not reported. Many other reports have been published about the mechanical characteristics of bonded interfaces in simulated (Mitchem, Terkla & Gronas, 1988; Prati, Pashley & Montanari, 1991; Davidson, Abdalla & de Gee, 1993; Paul & Scharer, 1994; Nikaido & others, 1995; Mitchem & Gronas, 1991; Gerhardt, Szep & Heideman, 1995; Krejci & others, 1994) or real (Pashley & others, 1993) physiological conditions, but, at the moment, the correlation between the sealing ability and the bond strength of the new polyacid-modified composite resins or the monocomponent DBAs has not been studied.

The objectives of this paper were to analyze the variations of the permeability of dentin after sealing with different materials immediately after insertion, to study the bond strength of their interfaces, and to find the correlation, if any, between both parameters.

METHODS AND MATERIALS

Surgically extracted sound third molars had their roots removed with a diamond disk, exposing the pulp chamber. The soft tissue was removed with cotton pliers, taking care not to touch the chamber

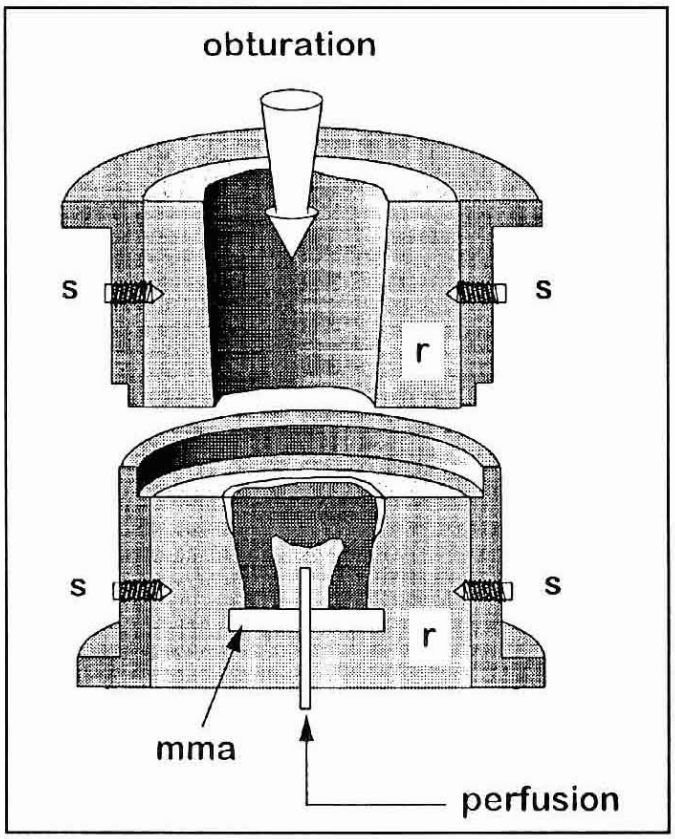

Figure 1. *Schematic hemisection of setup. mma = methylmethacrylate base; r = embedding resin; s = fixing, embedded screws. Obturation is made through upper matrix (white arrow) when the device is assembled.*

roof. With a commercial cyanoacrylate glue (Super Glue 3, Loctite, Madrid, Spain), a rectangular piece of methacrylate with a hole in its center was attached to the radicular portion of the crown segment (Figure 1). A metal tube was passed and sealed through a hole in the plexiglass, allowing the perfusion of each specimen. Occlusal enamel was ground away using 120-grit paper (Struers, Rodovre, Copenhagen, Denmark), thus exposing as much dentin as possible. The dentin and enamel areas were measured in each specimen with an image analyzer (VIDS IV, AMS, London, England, UK). This surface was finished with 600-grit paper. The specimens were embedded in a cylindrical device with a polyester resin (Cronolita 10700 + Activator 3015, Plastiform SA, Madrid, Spain). In the upper part of the assembly, a resin matrix was fabricated to confine the restorative materials. Through this matrix the restorative materials were inserted, once both sections of the assembly were properly positioned (Figure 1).

The teeth were connected to a perfusion system, at a pressure of 32.5 cm of distilled water through the metal tube into the pulp chamber. A 100±1 µL graduated micropipette was inserted between the tooth and the pressure reservoir. Once the system was stable, an air bubble was placed in the micropipette with the help of a microsyringe. The movement of this bubble permitted measurement of the fluid volume lost from the system through the cut dentin surface.

The fluid flow through each specimen was measured in µl/min for 30 minutes (preinsertion period), with readings approximately every 5 minutes. After this, the cut dentin surfaces were obturated through the resin matrix with the DBA and the restorative material, and the fluid flux was measured during a postinsertion period between 60 and 120 minutes, reading in a similar fashion as in the preinsertion period.

Each material was prepared following the manufacturer's instructions and placed in at least three increments, curing each one for 40 seconds (Translux C L, Kulzer, Wehrheim, Germany). Materials tested were: Compoglass (COM) with SCA, Tetric with Syntac SC (SYN) (Ivoclar/Vivadent, Schaan, Liechtenstein), Dyract with PSA (DYR), TPH Spectrum with Prime & Bond 2.0 (P&B) (DeTrey/ Dentsply, Konstanz, Germany), Z100 with Scotch Bond 1—named Single Bond in USA—(SB1) (3M Dental Products, St Paul, MN 55144) and Prodigy with OptiBond Solo (OPT) (Sybron/Kerr, Romulus, MI 48174).

In some materials (P&B, SYN, OPT, SB1) two parts of the postinsertion curve were defined (Figure 2). The first part was the first 30 minutes postinsertion; this part of the curve followed a logarithmic decline. Its slope was not considered in the calculations of the final fluid flow decreased, because it corresponded to the rehydration of the teeth following etching and air drying. Its slope is referred to in the text, results, and tables as the *immediate decrease*. The second part was linear, and calculations of the decrease in fluid flux (*final decrease*) were made using its slope.

In any period, the slope of the regression line of the data volume (µl) to data of time (minutes) was used as the parameter defining the fluid filtration.

Once the postobturation time had passed (120 minutes), tensile force was exerted over the entire restored surface (enamel and dentin) at a crosshead speed of 1 mm/min (H 5000M/79L, Houndsfield Test Equipments, Croydon, England, UK), in the direction perpendicular to the bonded surfaces.

There are some reports (Roderer & others, 1995; Fowler & others, 1992; Burrow & others, 1994) that measured the bond strength of several materials to enamel and dentin under the same conditions. The pooled ratio of enamel tensile bond strength to that of dentin was found to be 2.003. This relationship was taken into account in our calculations to assign the corresponding bond strength of resins to the different dental tissues.

ANOVA and Newman-Keuls tests were carried out to find if there were statistically significant differences among the results for each material on final fluid flow decrease and on tensile bond strengths (TBS).

Regression correlation of decrease in fluid flow versus TBS was calculated for P&B, SYN, DYR, and COM.

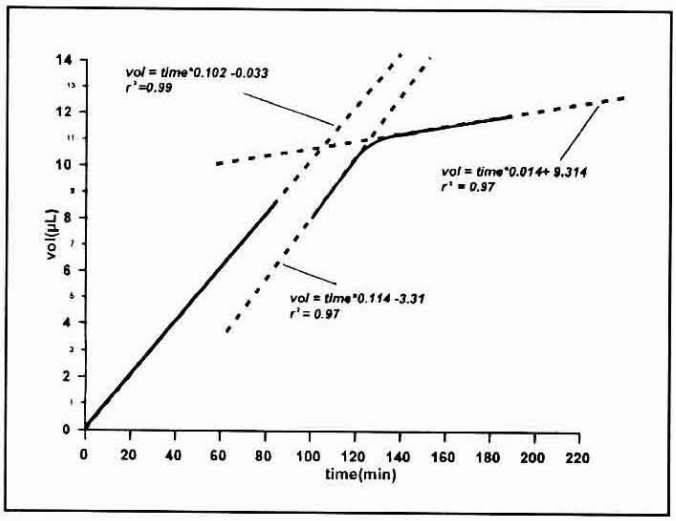

Figure 2. *Plotting of one sample of SB1, showing preinsertion, immediate, and final parts of postinsertion periods. The break in the line was due to the time of insertion of the material; note the slow decrease in fluid flow over 30 minutes postinsertion, until it reached a constant slope.*

Table 1. Reductions in Fluid Flow across Dentin Produced by Various Restorative Materials

		IMMEDIATE (0-120 minutes)			FINAL (30-120 minutes postinsertion)		
Material	**n**	**Mean**	***SD***	***SE***	**Mean**	***SD***	***SE***
COM	17	77.50	10.47	2.63	---	---	---
DYR	16	73.70	12.29	3.34	---	---	---
SYN	14	75.84	8.11	---	86.00	5.50	1.79
P&B	24	73.66	15.51	---	81.06	15.60	2.56
SB1	18	81.00	16.06	---	88.57	10.97	2.56
OPT	16	72.14	17.24	---	83.39	13.69	3.72

Values are percent reductions from the nonrestored values. A theoretically perfect seal would produce a 100% reduction in fluid flow.
COM = Compoglass with SCA; DYR = Dyract with PSA; SYN = Tetric with Syntac SC; P&B = TPH Spectrum with Prime & Bond 2.0; SB1 = Z100 with Scotchbond 1 (Single Bond); OPT = Prodigy with OptiBond Solo; n = number of specimens; SD = standard deviation; SE = standard error ($P < 0.001$). Mean ± SE ($P < 0.001$): Limits for % reduction in postinsertion fluid flow relative to preinsertion values with 99.9% confidence.

RESULTS

The Kolmogrov-Smirnof one-sample test was used to measure the amount by which the empirical cumulative distribution function differed from that of the filled distribution. No significance was found. The ANOVA and Newman-Keuls tests were employed to look for differences in the mean values of continuous quantitative variables.

Table 1 shows reductions in fluid flow across dentin produced by the restorative materials. Values are given as percent reductions from the nonrestored values in the various treatment groups, in the immediate and final postinsertion periods. ANOVA testing showed that there were statistically significant differences among the materials ($P < 0.05$). The Newman-Keuls test determined that the difference was: SB1 > DYR ($P < 0.01$) in the immediate group, with all other materials not statistically different.

Table 2 summarizes the TBS data (MPa) for the whole surface and that corresponding to dentin (details given below). ANOVA testing showed that there were statistically significant differences among the materials ($P < 0.01$). The Newman-Keuls test determined the differences were: P&B > DYR ($P < 0.001$), P&B > COM ($P < 0.01$), and P&B > SYN ($P < 0.05$).

Figures 3-6 show the plots of the decrease in final fluid flow versus total tensile bond strength with each regression line. In all cases, the correlation between fluid flow decrease and tensile bond strength was very low and statistically insignificant.

DISCUSSION

Fluid Flow Decrease (FFD)

There are many reports of variations in dentin permeability due to the interaction with different restorative materials or DBAs (Pashley & others, 1985; Pashley & Depew, 1986; Del Nero & others, 1994, 1997; Prati & others, 1992, 1994a; Hansen & others, 1993; Pagliarini & others, 1996; Déjou & others, 1996). In these reports, larger decreases in permeability occurred when the smear layer was eliminated. In the studies where smear layer was not removed, the percentage of permeability decrease was smaller, although many of the materials tested required some kind of etching. One explanation could be that the baseline was more unfavorable if the dentin was etched, because this procedure increased permeability. None of the published studies reports the creation of a perfect seal (i e, 100% decrease in fluid flow). Our work was in accordance with those previously published reports. Our results indicated a wide range in the decrease in permeability, which was highly variable, depending on the kind of material and the type of specimen used. Nevertheless, our results were in accordance with those of Terkla and others (1987), who used a similar tooth preparation.

Table 2. Dentin and Total Tensile Bond Strengths of the Various Restorative Materials (MPa)

		Dentin		Total		
Material	**n**	**Mean**	**SD**	**Mean**	**SD**	**SE**
COM	14	1.74	0.75	2.39	1.08	0.35
DYR	12	1.22	0.46	1.64	0.62	0.25
SYN	9	2.06	0.90	2.72	1.22	0.77
P&B	8	2.97	0.97	3.96	1.24	0.96

n = number of specimens; SD = standard deviation; SE = standard error ($P < 0.001$). Mean ± SE ($P < 0.001$): Limits for tensile bond strengths with 99.9% confidence.

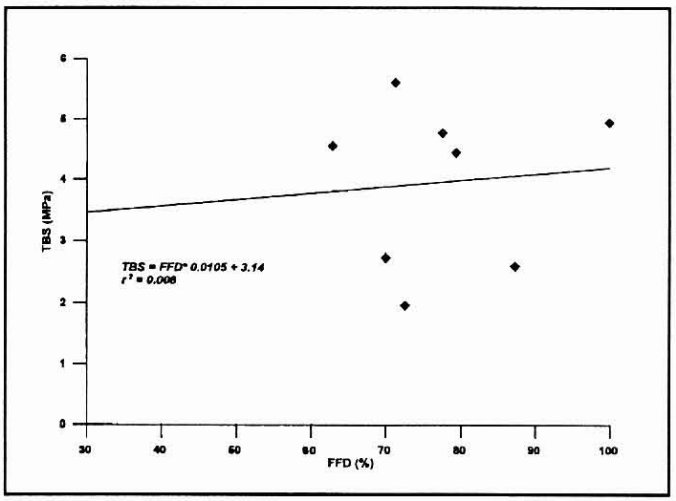

Figure 3. *Plot of final decrease of permeability (FFD) versus total bond strength (TBS) for P&B*

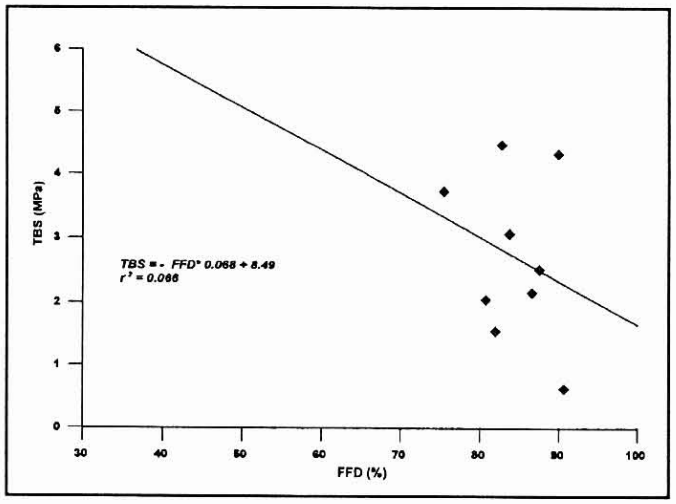

Figure 4. *Plot of final decrease of permeability (FFD) versus total bond strength (TBS) for SYN*

With our methodology it was not possible to reconcile the persistence of fluid perfusion after insertion of restorations with the very good clinical behavior of these materials (Pashley & Carvalho, 1997; Nicholson & Croll, 1997; Van Meerbeek & others, 1998). Generally, the quality of the interface between teeth and restorative materials has been studied using the leakage of dyes. However, the absence of dyes or, to be more precise, the lack of their detection, does not necessarily mean that the interface is hermetically sealed to water, which has a molecular weight of 18, as opposed to the much higher molecular weights of currently used dyes (ca 200-300; Pashley, 1997).

In our experimental design, the residual permeability of restorations may be due to fluid loss through the unsealed dental surfaces. In our experience, applying nail varnish to such surfaces reduces specimen permeability to a minimum percentage (unpublished data), but not to zero. It has to be remembered that when using dentin disks (Pashley & others, 1985; Hansen & others, 1993; Del Nero & others, 1997) the expected 100% seal may not be obtained, possibly due to fluid leakage across the lateral surfaces of the disks.

The behavior of the fluid flux seems to have different patterns depending on the material studied. For COM and DYR (both polyacid-modified resins) the pattern seemed to be strictly linear. For the other materials (P&B, SYN, OPT, SB1), which require total etch, it seemed to have two different patterns. The first part of the plotting may represent the rehydration of the dentin from the pulp chamber, and

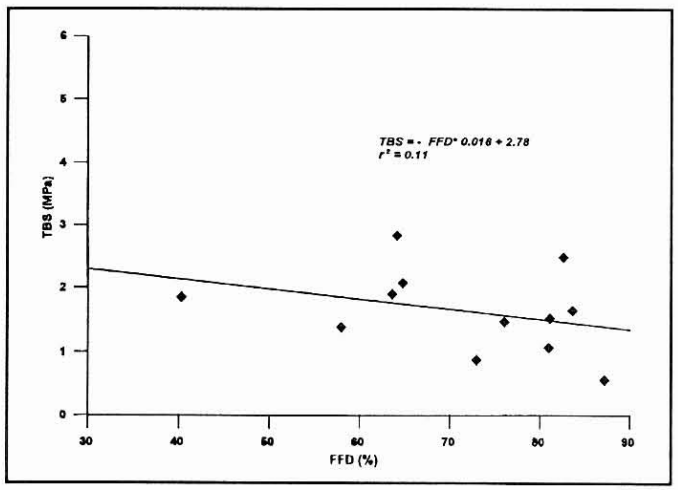

Figure 5. *Plot of final decrease of permeability (FFD) versus total bond strength (TNS) for DYR*

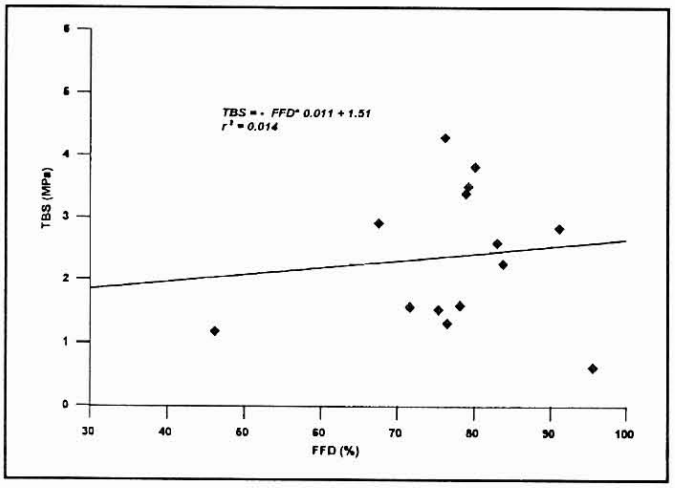

Figure 6. *Plot of final decrease of permeability (FFD) versus total bond strength (TBS) for COM*

does not mean, in our opinion, that there was fluid flow through the bonded interface.

Morphological studies have detected the existence of a gap between materials and dental tissues (Perdigao & others, 1996), but the absence of such a gap in the microscopic preparations does not guarantee either a perfect interphase, or that the materials are impermeable to fluids. Our methodology permits better discrimination and indicates that hermeticity does not exist. According to Prati, "There is invariably a gap between restoration and tooth . . . , allowing fluid flow" (Prati, 1994).

Water Sorption

A more likely explanation of the residual fluid flow resides in the chemical characteristics of the materials we used in this study. All of them are known to absorb water and this absorption can happen through the DBA layer, thus producing a false residual permeability. Yet such water absorption is an unappreciated phenomenon. Yap and Lee (1997) have measured the aqueous absorption of, among others, one of the composite resin materials we used in our study, Z100. They reported a water absorption, within 7 days, of 28.79 (SD = 1.165) µg of water/mm^3 of material, with an exposed area of 400.55 mm^2. Operating with their data, we can estimate that such material has an absorption of 7.1316E-6 µg of water/ mm^2 of exposed surface, per minute. Observations must be made about: (1) the rate of absorption not following a linear model because there must be a lag time before the material is saturated with water, and (2) when the absorption rate through the interface becomes equal to the evaporation rate through the exposed surfaces. When this equilibrium has been reached, the material will absorb water only to balance the evaporation rate. In our model, water absorption occurred through a surface covered with a DBA, a fact which will probably influence its rate.

Table 3. Total Volume of Water Absorptions of Materials after 25 Minutes

Material	µL H_2O/mm^2 of Exposed Surface
Spectrum	0.046
Prodigy	0.101
Z100	0.068
Dyract	0.091
Compoglass	0.065

Moreover, the cited work used overdried material, unlike ours, and submerged all surfaces of the samples in distilled water. In our model, water absorption could only occur through the DBA-dentin interface. It can be expected that, for the materials used in our study, the water absorption was probably higher than that reported by Yap and Lee (1997).

Our data (unpublished observations) showed that, in the first 25 minutes, some of the materials that we tested had extremely low equivalent water absorptions, as cited in Table 3.

Such values of water absorption do not seem to be responsible for the perfusion values that we recorded, as our measurements had a sensitivity of ±1 µL, and the system did not have enough sensitivity to perceive such small changes.

Tensile Bond Strength (TBS)

The values found for the resin bond strength were very low, which may well have been due to "the effect of the presence of defects and/or stress risers at the interface or in the substrate" (Sano & others, 1994). According to Griffith's theory (Griffith, 1920), it is more probable to find a defect that initiates the fracture in a larger area than in a smaller one. To support this idea, we have found cohesive fractures of dentin and restorative materials at very low apparent bond strengths (not included in the data), i e, < 5 MPa.

There was an enamel collar surrounding the dentin area, which contributed to the total bond strength. However, regardless of whether we used the total tooth surface or the dentin surface in the calculations, our values were very low.

Correlation of FFD/TBS

The correlation between reductions in fluid flow and bond strength were very poor for all the materials tested. We think this was due to the very low bond strength values that were found. According to Sano and others (1994) and Pashley (1997), if we use a material such as Clearfil Liner Bond II, the relation between TBS and the bonded surface (BS) should follow the formula $TBS = 58.8 - 27.9 \times \log_{10}(BS)$. For a bonded surface of 88.8 mm^2 (mean bonded surface area in our work), this equation would predict a TBS = 4.4 MPa. We think our results fit acceptably with such a prediction, taking into account the estimation error, the differences of materials, and the higher variability that occurs when the areas increase. In fact, our best-rated material (P&B) had a TBS of 3.96 MPa (Table 2). There was also almost a 40% decrease in the number of TBS samples (43) after the fluid flow tested 71 samples. The loss in specimens was due to the approximate loss of 75% of

the P&B specimens between these testing sequences. Specimens were discarded when any abnormality was detected at the moment the filtration device was assembled to the traction machine (almost exclusively "spontaneous" separation) or when, unintentionally, any of the parameters of the traction were not fulfilled properly (mainly crosshead speed).

Clinical Consequences

The clinical consequences of this work are that it may be impossible to create a hermetic seal in any restoration, at least with the materials tested. The fact that the clinical behavior of these materials is considered acceptable leads us to believe that a hermetic seal is neither necessary nor even possible. There will always be a passage of water, at least in an outward direction, that could be interpreted as an interchange of fluids towards and (probably) from the environment, through the restorative materials. It is the rate of such interchange that decreases but does not cease following restoration of cavities. Apparently this small fluid exchange is acceptable to the dentin-pulp complex. This may be a case where the sensitivity of a measuring device is so high that its detection is beyond clinical relevance (Pashley, 1997).

Nevertheless, the fact that there is a path for fluids through the interface means that restorative/adhesive materials are in a detrimental environment, although there would be no clinical evidence of such fluid flow. In this case, hydrolytic stability of materials becomes critical. The best interface (i e, maximum tightness) is achieved in the first stages of adhesion, and it can only become worse with time, especially in the oral environment.

Although we could not demonstrate the relationship between final decrease of fluid flow and TBS, it does not seem likely to us that higher TBSs would produce fluid flow cessation.

Unfortunately, it is impossible to select minimum bonding surfaces in a clinical situation, because they are determined by the type of cavity preparation, the skills of the operator, and the extent of the caries process. Recently, the bonding surface areas of different cavities were measured (de la Macorra & Gómez-Fernández, 1996). Class 1 cavities have a mean bonded surface area of 39.94 ± 7.54 mm^2, class 2 cavities 76.38 ± 24.61 mm^2, and class 5 erosions of 17.75 ± 5.10 mm^2 ($P < 0.05$). In such large areas, one can expect that TBS would be lower than those that are measured using smaller surfaces.

The mechanical stress on clinically bonded class 5 cavity surfaces is not perpendicular to all surfaces simultaneously, although the restored cervical erosions are closest to a perpendicular stress. The above cited formula (Sano & others, 1994) predicted a TBS of about 24 MPa for a bonded surface area of 17.7 mm^2.

CONCLUSIONS

None of the materials tested in this study produced complete cessation of fluid flow. The residual permeability that was found was interpreted as due to the passage of water vapor through the material and the adhesive.

Tensile bond strength values were very low, which was consistent with the predictions of low bond strengths in specimens with very large surface areas.

No correlation was found between tensile bond strength and the ability of any material to seal dentin.

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